

CEMENT AND LIME MANUFACTURE

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Testing Cement and Concrete.

PAPERS READ AT THE CONGRESS OF THE INTERNATIONAL
ASSOCIATION FOR TESTING MATERIALS.

A VERY large number of papers was presented to the International Congress of the International Association for Testing Materials held in London from April 19 to 24 last. Abstracts of some of the papers relating to the testing of cement and concrete are given in the following. Abstracts of other papers will be given in subsequent issues of this journal.

TESTING CEMENT AND CONCRETE BY ELECTRIC HEATING.

By **Albert Brund.**

HÄRNÖSAND, SWEDEN.

In 1928 the author and H. Bohlin evolved a method of accelerating the curing of cement and concrete specimens by means of electric heating. Tests showed that specimens heated for about 8 hours to a maximum temperature of 80 deg. C. and then cooled to room temperature have a compressive strength amounting to about 75 per cent. of that attained by similar test pieces after curing for 28 days under natural conditions. These results have been verified by Réthy (Moscow), and it was obvious that electric heating would be advantageous in determining the strength after 28 days at about 24 hours.

When alternating current is fed to a fresh cement or concrete mixture, heat is generated in the paste thus accelerating the reaction between water and cement in the mixture. It was found that the resistance decreased during the first period of hardening, up to the beginning of binding, where it reached a minimum, and increased during subsequent heating as the paste hardened. The rate of curing was uniform throughout the mixture. The reason is that if at some point of the mass the current develops more heat than elsewhere so that curing progresses further at that point, then its electrical resistance increases with the result that the current deviates to some other point where curing has not yet

progressed so far and where the resistance is smaller. In this way the heat development and curing are automatically regulated, resulting in a uniform distribution of heat and moisture. Specimens produced in this manner will be of thoroughly uniform hardness, so far as this is possible with present mixing methods, so that a considerable reduction in the variation of strength obtained from tests ensues. In general, the deviations of the strength figures from the mean value were below 2 per cent.

To determine the strength which a cement or concrete mixture will have after a definite period of time, the mixture is placed in a metal box lined with non-conducting material. The size of the box is such that the specimens have standard dimensions. Thin metal plates serving as electrodes are inserted between the sample paste and the insulating material on two opposite surfaces. A tight cover is screwed on the box so that it resembles an autoclave. A thermometer is placed in the box in such a manner as not to damage the mixture, or a thermo-electric element may be introduced. The current is regulated by rheostats. A voltmeter and an ampere-meter are added if the resistance is to be measured.

CONTROLLING THE HEAT OF HYDRATION OF CEMENTS.

By P. H. Bates.

NATIONAL BUREAU OF STANDARDS, U.S.A.

Three methods for the production of cements with low heats of hydration have been suggested. According to the method mostly employed the usual composition is changed by the reduction of the alumina and lime content, accompanied by a large increase in the amount of iron oxide and a lesser increase in the silica, or by a large increase in silica and a reduction in the amount of the other three major oxides. The second method is based upon "heat-treating" the clinker, and the third upon a partial hydration of normal cements.

The procedure of altering the heat of hydration by limitations of composition is based upon the assumptions that there are certain compounds present in cement, that the heat of hydration (as well as strength) of the cement is the sum of that of each compound, and that there are negligible secondary reactions between the hydrated compounds. The heats of hydration of the pure compounds have been determined, and the most active of these are found to be tricalcium aluminate and tricalcium silicate. Hence specifications have been adopted limiting the permissible amounts of these compounds. But data have accumulated which question the correctness of the assumption that some of these compounds are either present or present in the amounts calculated from the hypotheses. After making due allowance for the effect of adsorbed moisture and size of cement grain, it has been noted that calculated and observed heats (as well as strengths) do not agree and that cements of almost identical calculated composition may show large differences in heats or strengths. This is illustrated in Table I, in which the pair in any group should be compared.

Undoubtedly these discrepancies are due to the lack of validity of the assumptions that during the formation of the clinker and the cooling complete crystallisa-

TABLE I.

| Cement group. | | A | | B | | C | | D | | E | |
|--------------------------------|---|------|------|------|------|------|------|------|------|------|------|
| Per cent. | 3CaO.SiO ₂ | 20 | 20 | 25 | 25 | 19 | 18 | 51 | 48 | 41 | 42 |
| " " | 2CaO.SiO ₂ | 53 | 51 | 45 | 48 | 51 | 52 | 25 | 29 | 33 | 36 |
| " " | 3CaO.Al ₂ O ₃ | 6 | 6 | 6 | 5 | 5 | 4 | 5 | 6 | 7 | 6 |
| " " | 4CaO.Al ₂ O ₃ | 16 | 15 | 16 | 16 | 17 | 17 | 13 | 12 | 13 | 12 |
| Cal. per g. | 7 day { Calculated | 52 | 52 | 56 | 54 | 48 | 45 | — | — | — | — |
| | { Observed | 60 | 53 | 53 | 62 | 59 | 49 | — | — | — | — |
| | 28 day { Calculated | 67 | 67 | 70 | 70 | 63 | 60 | — | — | — | — |
| | { Observed | 68 | 61 | 62 | 69 | 66 | 57 | — | — | — | — |
| Compressive strength / 7 days | | 2070 | 1360 | 1630 | 2150 | 1530 | 1220 | 4430 | 1890 | 3420 | 2010 |
| mortars, lb. per sq. in.* { 28 | | 4420 | 3180 | 3010 | 4160 | 4900 | 3110 | 6500 | 4770 | 5800 | 4620 |

* 1 lb. per sq. in. = 0.0703 kg. per sq. cm.

tion has been obtained. But Rankin and Wright¹ have indicated in their phase equilibrium studies of the system CaO-Al₂O₃-SiO₂ that conditions may obtain under which complete crystallisation would not take place. Lea and Desch have studied this further² in the system CaO-Al₂O₃-Fe₂O₃-SiO₂. According to the amount of glass which may be present and its properties, the properties of the finished cements may vary considerably from those deduced from assumptions that no glass is present.

Further, if the amount of glass may induce marked differences in physical properties, it would follow that the physical properties could be altered, without altering the composition, by those heating (or cooling) procedures that would change the relative amounts of glass and crystallised compounds. Unknown and uncontrolled changes in kiln or cooler operations may induce such changes at times and may explain the unsought changes in physical characteristics of cements from the same mill. In view of the possibilities of lowering the heats of hydration (and altering other properties) by "heat treatment" of usual normal clinker, there have been developed methods involving reheating and recooling the clinker under certain predetermined optimum temperatures and conditions. These procedures have not been very adequately described, but they seem to be based upon the reduction of the glass content through the development of more crystalline material. This is in accord with the assumption that the latent heat of crystallisation of the glass would increase the heat of solution of a cement over that which would obtain if the glass had crystallised. Hence the "crystallised" cements would have lower heats of hydration and lower early strength than the glassy cements. The few data available show that "heat treatment" following clinkering may reduce the heat of hydration by 25 per cent. and the early strength by slightly less percentages.

¹ Rankin and Wright, "The Ternary System CaO-Al₂O₃-SiO₂," *Amer. J. Sci.*, Jan. 1915, vol. 39.

² Lea and Desch, "The Quaternary System CaO-Al₂O₃-Fe₂O₃-SiO₂," *Building Research Paper* 16, H.M. Stationery Office.

A recent publication³ gives results which may be obtained by the third method. The prehydration was carried out by treating ground cement in an atmosphere of steam at 100 deg. C. in a rotating oven until the ignition loss was increased to approximately 3 per cent. in one case and 5 per cent. in another. The cement was then reground to the original fineness, after which the heat of hydration and the compressive strength of the cement, cured at 21 deg. C. and 65.6 deg. C., were determined, as well as the expansion of mortar bars stored in water and in a 2 per cent. solution of sodium sulphate.

The data show that the average reduction in heats of hydration for all the cements at 7 and 28 days was about 6 calories, and at one year 4 calories per g. for each per cent. increase in ignition loss. At 7 and 28 days all standard Portland cements had heats of hydration within the limits specified for Boulder Dam, excepting those with 3 per cent. prehydration cured for 28 days. After curing at 21 deg. C. the strength of the prehydrated cements exceeded that attained by Boulder Dam cements cured for 7 and 28 days, but was less when cured for a year. Cured at 65.6 deg. C. the prehydrated cements developed less strength than the Boulder Dam type at all ages. The resistance to sulphate action was increased by the prehydration treatment.

TESTS OF WATERPROOFING MATERIALS.

By J. Løventhal.

DIRECTOR OF THE NATIONAL TESTING INSTITUTE, COPENHAGEN.

The Danish National Institute for Testing Materials has carried out tests of commercial waterproofing materials for concrete. The test-specimens generally employed were circular plates of mortar, 12.5 cm. in diameter and 2.5 cm. thick; the test can be carried out just as well with concrete cylinders 10 cm. in height.

Two or three plates were made from a mixture of 1 part cement to 3 parts sand (by weight), sufficient water being added to ensure that the consistency of the mortar was plastic. The waterproofing material was added as given in the instructions for its use. A similar number of plates, for comparison, was made from the same materials in the same proportions and of the same consistency as the others, but no waterproofing was added. The water-cement-ratio varied from 0.54 to 0.64 according to the cement and proofing medium used. The sand was fairly fine, and it was found that, with testing plates made without waterproofing, some percolation occurred during the permeability test.

The amount of water percolating through the unproofed testing plates varied somewhat owing to the use of different kinds of cement. For this reason it was necessary in every case to make testing plates without waterproofing for purposes of comparison. The plates were kept in moist air for 24 hours and then removed from the moulds, wrapped in damp cloths, and stored for 6 days. After this they

³ Hornibrook, Kalousek and Jumper, "Effect of Partial Prehydration and Different Curing Temperature on some of the Properties of Cement and Concrete." *National Bureau of Standards, Research Paper 887*, 1936, vol. 16, pp. 487-509.

were kept for three weeks in air at 15 to 20 deg. C. and 50 to 60 per cent. humidity. At the end of this period both plane surfaces of the plates were cleaned with sandpaper to remove any deposit of neat cement that might have accumulated there, coated with asphalt, and subjected to water-pressures of 1, 2, 3, 4, 10, 20, 30, and 40 m. Each pressure was maintained for 24 hours, and the amount of water percolating was measured. All the waterproofing materials examined were mixed in the mortar or concrete. The liquids and pastes were mixed with the gauging water, while the powders were, as a rule, mixed with the cement.

The tests showed very great differences in the value of the various waterproofing materials, and gave different results with different water pressures. The liquids and the pastes seemed, as a rule, to be more effective than the powders. None of the materials made the mortar absolutely proof against the higher pressures, whilst some of them made the mortar impermeable at a pressure of 2 to 3 m. only. About the same results were obtained with different samples of the same sort of waterproofing and with different kinds of cement. Experiments were also made with mortar mixed in various proportions to ascertain the effect of increasing the cement content. Table I gives some examples of the results. Materials X and Y in the table are, respectively, the best and the poorest of those examined. The table shows that the addition of a suitable amount of cement makes the mortar much more impermeable, and that complete impermeability, practically speaking, can in this way be obtained in certain cases. As the addition of cement, as well as increasing the impermeability, increases the strength, it is thus preferable to the addition of a waterproofing material in cases where the shrinkage, caused by the richer mortar, does not prevent the use of this method.

TABLE I.

| Material. | | Pro- portions | Percolation, in c.c., with Water-pressure of | | | | | | | |
|-------------|--------------------------------|------------------|--|------|------|------|-------|-------|-------|-------|
| | | | 1 m. | 2 m. | 3 m. | 4 m. | 10 m. | 20 m. | 30 m. | 40 m. |
| Cement A | Without water- proofing. | 1 : 3 | 32 | 64 | 100 | 132 | 402 | 648 | 1022 | 1158 |
| | With water- proofing X | 1 : 3 | 0 | 0 | 0* | 6 | 30 | 81 | 135 | 172 |
| Cement B | Without water- proofing. | 1 : 3 | 1.8 | 12 | 16 | 20 | 70 | 139 | 183 | 230 |
| | With water- proofing Y | 1 : 3 | 1.2 | 13 | 21 | 28 | 89 | 170 | 214 | 256 |
| Cement C | Without water- proofing | 1 : 3 | 6.6 | 20 | 30 | 39 | 151 | 247 | 319 | 431 |
| | | 1 : 2½ | 0 | 0 | 0 | 0 | 0 | 0 | 0* | 0* |
| | | 1 : 2 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Cement D | Without water- proofing | 1 : 3 | 0* | 0* | 1.1 | 3.6 | 15 | 19 | 25 | 29 |
| | | 1 : 2½ | 0 | 0 | 0* | 0* | 1.6 | 5.4 | 5.1 | 4.8 |
| | | 1 : 2 | 0 | 0 | 0 | 0 | 0* | 0.5 | 1.8 | 1.8 |

* Damp spots appeared on the underside of the plates, but there was no percolation.

TESTS ON PLASTIC MORTARS IN RELATION TO OTHER TESTS.**By Dr. Eng. A. Perfetti.**

ROYAL EXPERIMENTAL INSTITUTE OF COMMUNICATIONS, ROME, ITALY.

Tests have been made on thirteen Italian aluminous, Portland, pozzolanic and blast-furnace cements. Test pieces were made for tensile and crushing strength tests with rammed mortars according to Italian specifications, others for tensile and crushing strength of plastic mortars in Italian forms, others for crushing strength according to French specifications, and others for bending tests according to Swiss specifications. Test-pieces were made by the French and Swiss methods with standard monogranular sand as well as with "Haegermann" sand. For all the tests siliceous sand from Torre del Lago was used. Cubes of concrete were made, with sides 16 cm. long, from these cements. The aggregate consisted of gravel and sand divided in eight classes as used by Abrams for the determination of the modulus of fineness. The proportions were in accordance with the Fuller curve. For every cubic metre of aggregate 300 kg. of cement were used and the ratio of cement to water was equal to two. The test pieces were broken after 3, 7 and 28 days. For all the types of mortars six test pieces were made and the average of the four best results taken; for the concretes, series of four cubes were made and the average of the three best results taken.

From the results of these tests the following conclusions may be drawn:

- (1) In acceptance tests for cements, rammed mortars do not offer any advantage over plastic mortars.
- (2) The relative order attributed to all types of cements when testing plastic mortars with Italian test pieces is almost the same as that obtained when testing rammed mortars.
- (3) The relative order attributed to the different types of test pieces of plastic mortars differ one from another and in some cases are inverted.
- (4) The use of "Haegermann" sand causes noticeable differences in the results given by the cements examined.
- (5) The results obtained from bending tests using monogranular sand agree fairly well with those obtained from tensile tests on plastic mortars also using monogranular sand.
- (6) The results obtained from the tests on concretes lead to a relative order differing from that obtained with plastic mortars.
- (7) The maximum and minimum differences between extreme values and average values are much smaller for concretes than for mortars.

The following considerations may be deduced from the experiments. Cement is required to bind together different size pieces of aggregate. Cement in monogranular mortars has a very different function from that in concrete. In bigranular sand, like "Haegermann" sand, the structural conditions of the cementing paste are much nearer to those of concrete. This is why the results obtained with plastic or rammed mortars do not always reflect the aptitude of cements for the production of good concretes. It is not possible to obtain an exact evaluation of a cement by test on mortars; evaluations made on the basis

of tests of neat pastes are still more incorrect. For the evaluation of cements, tests on concretes should be made. This does not offer any great difficulty, as it is easy in practice to use an aggregate which is always the same in quality and grain characteristics, and to keep constant the other characteristics which influence strength so that the only variable item may be the cement.

In tests of concrete, more uniform results are obtained than by the other methods of testing.

If the use of standard concrete for tests of cements should not be accepted the next best method would be the test with plastic mortar made with monogranular sand with crushing-strength test pieces according to the French method and test pieces for bending tests according to the Swiss method. The simple tensile-strength test may be omitted, as the bending test is an adequate substitute.

STRENGTH TESTS FOR HIGH-ALUMINA CEMENT.

By W. H. Glanville, D.Sc., Ph.D., M.Inst.C.E.

A committee of the British Standards Institution has had under consideration the issue of a standard specification for high-alumina cement, and a considerable amount of experimental work has been carried out at the Building Research Station. It was felt desirable to include a strength test which would produce reliable results and form a guide to the behaviour of the cement in concrete. Tests showed that the tensile test was unsatisfactory from both points of view: the divergence between different laboratories and different gaugers showed large discrepancies and the tensile strengths bore little or no relation to the strength of concrete. Research was also made into testing methods for Portland cements. One method investigated entailed the use of a compression cube of 1:3 standard sand mortar, using 12½ per cent. of water by weight, finger-pressed into the mould. The results of these tests showed a very close relation to the results of tests on 1:2:4 concrete with a water-cement-ratio of 0.60; in fact, for all practical purposes the strengths could be regarded as identical. In order to examine the applicability of the test to high-alumina cement it was used for every batch of cement delivered to the Building Research Station. The results are shown in *Fig. 1*. The relation between the compressive strengths of concrete and mortar is as close as can reasonably be expected and the compressive strength of 1:3 mortar using 12½ per cent. of water is a useful index to the strength of concrete. Further tests have also been made by blending a cement intentionally produced of low strength with a good quality material. Throughout the range, satisfactory results were obtained.

The next step again followed that used for Portland cements, namely, the use of vibration. Two types of vibrators were used, a low-frequency machine running at 1,000 revolutions per minute with an amplitude of 0.1 in. and an acceleration of 2.83 g., and a high-frequency machine running at 12,800 revolutions per minute, with an amplitude of 0.00214 in. and an acceleration of 10 g. Both tests gave uniform results and good agreement with concrete strength.

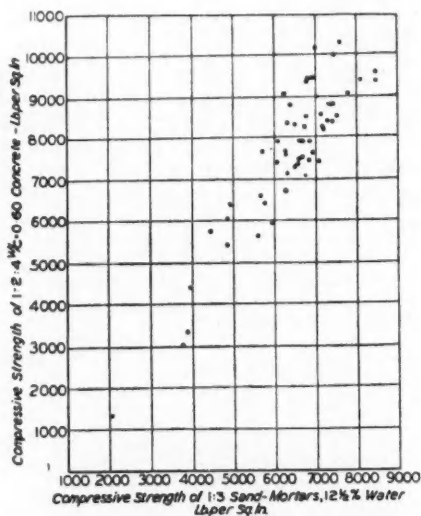


Fig. 1.—Relation between Crushing Strength of Concrete and Crushing Strength of Sand Mortar.

With the high-frequency vibration it was found possible to reduce the water content to 10 per cent., thus enabling higher strength figures to be obtained corresponding to the strength of 1 : 2 : 4 concrete with a water-cement-ratio of about 0.45. Tests by gaugers from various laboratories using the high-frequency vibrator show that the greatest variation from the mean is much better than anything experienced before. The results show that personal variations in methods of mixing, mould-filling and compacting have been almost eliminated.

METHODS OF TESTING CEMENTS FOR LARGE DAMS.

By **B. Hellström, M.Inst.C.E., M.Am.Soc.C.E.**

CHAIRMAN OF THE INTERNATIONAL SUB-COMMITTEE ON SPECIAL CEMENTS
FOR LARGE DAMS.

The International Commission on Large Dams in 1934 formed an International Sub-Committee on Special Cements, comprising representatives of Belgium, Czechoslovakia, France, Germany, Great Britain, India, Japan, Norway, Sweden, Switzerland and U.S.A. The Sub-Committee submitted an interim report to the Second Congress of the International Commission on Large Dams held in Washington in September 1936, and this paper contains a short summary of this report.

Heat of Hydration.—The more important methods of determining the heat of hydration have been dealt with in a report which Dr. Lea presented to the Congress, and the interim report gives a summary of the methods investigated

by the Sub-Committee. The Committee considers that the full adiabatic method giving thermal data for concrete actually used would be preferable. The method recommended was worked out by the Building Research Station in Great Britain for the British Committee on Special Cements. Comparative tests of this method have been carried out at different laboratories in England, Sweden and Norway, and it was found that the results are sufficiently accurate for a routine method. An investigation of the method of observing the temperature rise of cement mortar in thermos flasks was also carried out. Although it is considered that this method is worth consideration as a standard method of testing, it appears that further tests are required before a definite opinion can be expressed.

Shrinkage.—The Sub-Committee considered that tests on hydrated cement would give values of shrinkage of another magnitude than tests on concrete, while, on the other hand, tests on concrete were slower and would be difficult to standardise. It is, therefore, considered that tests on cement mortar would provide the most practical means of testing shrinkage. The Sub-Committee has been studying a German method of testing shrinkage which appears worth consideration, and a description of this method is given in Appendix 6 of the interim Report.

Permeability.—The Sub-Committee has limited its activities in respect of permeability to submitting certain recommendations which should be applicable to any test method that may be preferred. Testing equipment used in various laboratories may be conveniently classified as follows: (a) Apparatus providing for a test specimen with two free surfaces, one for entrance and one for escape of the water. The total quantity of water penetrating the specimen enters through one surface and escapes through the other. (b) Apparatus providing for a test specimen of which all surfaces are free but a limited area of one surface is reserved for the entrance of water, and a limited area of another surface for its escape. The Sub-Committee believes it would be most suitable to use the first method. Two principal methods of testing were considered: (A) Determination of the minimum amount of cement per unit volume of finished concrete which, under fixed test conditions, gives impermeable concrete. (B) The determination of the permeability of concrete on different cements by using a test specimen with the same amount of cement per unit volume of finished concrete. The comparison of the different cements according to (A) is obtained on the basis of the minimum amount of cement required to obtain impermeable concrete, while according to (B) the basis of comparison is the amount of percolating water. Although the first method is somewhat more expensive than the second, it seems preferable to use this method whenever possible. As certain special cements may have a slow rate of hardening, it is found desirable to allow ample time for curing so as to obtain a fair comparison between different cements, and a comparatively long time of curing in water (25 to 53 days) with subsequent curing in moist air (at least 14 days) is recommended.

General.—At the executive meeting of the International Commission on Large Dams in 1936 the methods for testing heat of hydration, action on cement

of water percolating through concrete, and permeability, as specified in Appendixes 2, 5 and 7 of the Interim Report, were approved as tentative standard testing methods.

TESTING POZZOLANIC CEMENTS.

By F. M. Lea, D.Sc., F.I.C.

BUILDING RESEARCH STATION.

The testing of blended cements containing a mixture of pozzolana with Portland cement raises a number of special problems. In such cements the Portland cement fraction hydrates and develops strength at the usual rates characteristic of this material, but the reaction of the pozzolana is slower and more gradual. The strength developed at ages of 7, and even 28, days by pozzolanic cements depends primarily on the amount and rate of hardening of the Portland cement present and only secondarily on the pozzolana. The ultimate properties, such as the strength at long ages and the resistance to attack by sulphate-bearing waters, however, depend very markedly on the pozzolana.

As alternative methods for testing pozzolanic cements, one may either test the blended cement or require separate tests on the pozzolana and Portland cement constituents. The former method is adopted in certain national specifications which cover pozzolanic cements as well as Portland and other types. Though the application of the same strength tests as are used for Portland cement will, provided the test mortar is of plastic and not dry consistency, afford a check on the rate of strength development at early ages of a pozzolanic cement, it affords no test of the quality of the pozzolana present. The method of independent testing of the pozzolana and Portland cement constituents has been adopted in the specification¹ for pozzolanic cements for Bonneville Dam on the Columbia river, U.S.A., and is also given in the German specification for trass. In both cases the pozzolana is tested in a lime mortar. Although lime mortar tests are of value when considering lime-pozzolana mixes they do not appear to afford a satisfactory indication of the value of a pozzolana for use in mixtures with Portland cement.

Specification tests for pozzolanic cements should afford a measure of the strength which may be expected at early ages and also of the quality of the pozzolana. Investigations on pozzolanic cements have been in progress at the Building Research Station over the last eight years. In the more recent work particular attention has been paid to methods of test suitable for use in a specification.

The substitution of pozzolana for Portland cement reduces the strength of concrete at early ages but, as has been shown by much previous work, no corresponding reduction is found in the strength of standard mortars of dry consistency. Tests on such mortars are therefore misleading. Mortars of plastic

¹ Savage, J. L. (U.S. Department of the Interior) "Special Cement for Mass Concrete." Report presented at the Second Congress of the International Commission on Large Dams, Washington, D.C., 1936, p. 161.

consistency, corresponding to water contents of 11 per cent. or over in 1:3 cement standard-sand mortars, offer a much improved indication of the strength of concrete at similar ages. This is illustrated by the data given in Table I. Pozzolana *A* was a material of poor quality and pozzolanas *B* and *C* materials of good quality, as shown by long-age strength and sulphate-resistance tests.

The water required for a mortar of standard plastic consistency may be conveniently determined by a method similar to that described by Feret at the Brussels meeting in 1906 of the International Association for Testing Materials. The test is carried out on the mortar using a Vicat plunger and mould as described in the B.S.S. for Portland Cement (No. 12, 1931) but adding an additional load of 500 or 2,000 g. to the plunger and determining the water content required

TABLE I.—*Comparison of Strength Tests on Pozzolan Cements.* Strengths of cements composed of 60 per cent. Portland cement, 40 per cent. pozzolana by weight expressed as a percentage of the strength of the unsubstituted Portland cement in the same test and at the same age. All test specimens stored in water at 18 deg. C.

| | | Pozzolana. | | | |
|---|----------|------------|----|-----|-----|
| | | None | A | B | C |
| Tensile strength 1 : 3 dry consistency mortars (8% water) | 3 days | 100 | 75 | 86 | 89 |
| | 7 " | 100 | 81 | 89 | 93 |
| | 28 " | 100 | 95 | 104 | 104 |
| Tensile strength 1 : 3 plastic* consistency mortars. | 7 " | 100 | 51 | 59 | 59 |
| | 28 " | 100 | 61 | 66 | 74 |
| Compressive strength 1 : 2 : 4 concrete. Water content 60% by weight of cement. | 7 " | 100 | 57 | 59 | 55 |
| | 28 " | 100 | 60 | 67 | 60 |
| | 6 months | 100 | 67 | 75 | 84 |
| | 1 year | 100 | 73 | 94 | 94 |

* Consistency as determined by Vicat plunger test with 500 g. additional load.

for the plunger to penetrate to a distance of 10 mm. from the base of the mould. Comparative tests in three different laboratories have shown that the method of mixing the mortar and filling the mould can be sufficiently closely defined to give adequately reproducible results. For Portland cement the water content obtained corresponds to about 12.5 per cent. by weight of the dry mortar when 500 g. additional load is used, and 11.0 per cent. with 2,000 g. additional load. With different pozzolanic cements the values vary from a little below these figures to about 2 per cent. higher.

Compression tests on pozzolana mortars of dry consistency, though less misleading than tensile tests, still appear unsatisfactory. Compressive tests on plastic mortars may eventually be preferred to tensile tests, but adequate data are not yet available.

The strength tests at short ages do not differentiate between pozzolanic cements containing good and poor pozzolanas, since the strength obtained may be varied by changes in the proportion and rate of hardening of the Portland

cement present. Additional tests are therefore required. Investigations now in progress indicate that a satisfactory test of the quality of the pozzolana present may be obtained from the strengths developed in two sets of plastic mortar briquettes, one cured for one day in moist air at 18 deg. C. and two days in water at 18 deg. C., and the other cured one day in moist air at 18 deg. C., followed by 24 hours in water at 50 deg. C. and then one day in water at 18 deg. C. The ratio of the differences between the strengths developed under these two conditions to the sum of the strengths [i.e. (50 deg. strength—18 deg. strength)/(50 deg. strength + 18 deg. strength)] has been found to show a fair correlation with the resistance of pozzolanic cement mortars to attack by magnesium and sodium sulphate solutions and to the strength increase at long ages. Pozzolanic cements containing poor pozzolanas, or too low a proportion of a good pozzolana to have a high resistance to sulphate attack, and also Portland cements, give values for this ratio below 0.25 to 0.30, whereas satisfactory pozzolanic cements show values above these figures. Another test which has been found of some value is a form of lime extraction test on the set neat cement. By a combination of minimum strength requirements at 7 and 28 days with the tests discussed and with the inclusion of the normal requirements for setting time, soundness, etc., a reliable form of specification for pozzolanic cements seems feasible.

The Specific Surface of Cement.

By W. HORNKE.

IN Germany the fineness of cements is judged according to the residue on various sieves. For this purpose the chief figure used is the residue on the DIN sieve No. 0.09 (4,900 meshes per square centimetre); for high-strength cements the DIN sieve No. 0.06 (10,000 meshes per square centimetre) is used. Writing in *Tonindustrie Zeitung* (1936, p. 911), W. Hornke says that apart from the many errors inherent in such a process it does not give sufficient insight into the particle distribution of the cements. The portions which are smaller than $88\ \mu$ passing the DIN sieve No. 0.09, or smaller than $60\ \mu$ passing DIN sieve No. 0.06, cannot be further divided by sieving, but these fine portions influence the character of the cement a great deal. The surface, and therefore the speed of reaction, rises with increasing fineness. Hence arises the need for determining the specific surface, or at least the particle size distribution, of the cement. Of the many suggestions for the determination of the particle size distribution only a few can be carried out in practice.

The two principal methods, namely, air elutriation and sedimentation, both depend upon the varying speeds of fall of fine particles calculated according to Stokes's formula. In the case of air elutriation a definite air velocity removes a certain portion of the fine particles in the specimen and the residue is weighed; according to the velocity of the air it is possible to obtain individual particle sizes. The sedimentation method depends on the theory that in a mixture of cement and a liquid (e.g., cement and alcohol) the large particles fall faster than the small. According to Stokes's Law the minimum size of particle can be calculated which will have fallen below any given cross section of a column of the mixture at a given instant. If a sample is taken from this section it will contain all the small particles only. Andreasen¹ has designed such a sedimentation apparatus which is used in the cement industry and which will allow such a sample to be taken.

However, these methods present many difficulties. One source of error is that in the calculation of the time of fall spherical particles are assumed, whereas in practice the particles are of all shapes. If a large number of fractions is required the test takes a very long time. The sedimentation test, which is carried out quickly, does not give a complete analysis. The complete dispersion of the material in the sedimentation fluid is very difficult to attain, and is not always complete even if peptising agents, e.g., calcium chloride, are used. If a cement-alcohol mixture is used the time of fall of the large particles is so short that an accurate determination is not possible. On the other hand, if ethylene glycol is used, as is often recommended, a longer time of fall is obtained, but the separation of the cement from the liquid offers great difficulties and makes the determination inaccurate.

Lately, Dr. Haegermann² has devised a simple process by which the error,

¹ Andreasen, *Ber. d. DKG*, 1930, Heft 5.

² Haegermann, *V. DPZF. Protokoll.*, 1933.

when it is not too large, can be avoided. In a long series of tests he found that the fineness curves, which were obtained from the weight of the residue or the amount which went through in relation to the particle size, were uniform (Fig. 2). From a large number of empirical values shown graphically in Fig. 1, Haegermann was able to draw the curves in Fig. 2, which allow the complete particle distribution of a cement to be obtained when only one fraction has been determined. From these curves, for example, one can read off that a cement with 50 per cent. of the fraction 0 to 30μ contains about 20 per cent. of the fraction 0 to 10μ , or 60 per cent. of the fraction 0 to 40μ , and so on.

Haegermann's sedimentation apparatus allows the portion between 0 to 30μ

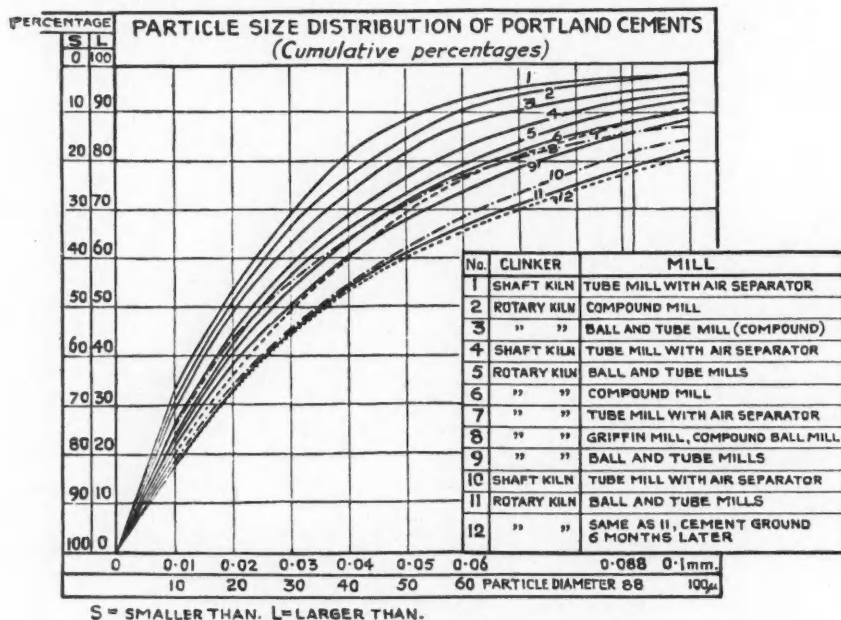


Fig. 1.—Particle Size Distribution of Cements.

to be obtained in a comparatively short time. It also allows the residue on the DIN sieve No. 0.09 (4,900 meshes per square centimetre) to be deduced very quickly. Haegermann³ proposed that the fineness of the cement should be characterised by these two figures.

In 1928, Kühl and Tokune⁴ published a method of calculation from which the specific surface of the cement could be obtained from the values of the individual particle sizes, and characterised the cement by this single figure. It is obvious that such a method of calculation which takes into account only the

³ Haegermann, *Zement*, 1936.

⁴ Kühl und Tokune, *Zement*, No. 8, p. 301 (1928).

individual particles can give only one point of view on account of the impossibility of determining the sizes smaller than 10μ . The same applies to Haegermann's method of estimation of fineness, since the constant particle size distribution is only a simplifying assumption and has not been established with certainty.

It is on account of this that the direct estimation of the specific surface is the true criterion. For some time the method of Witte⁵ has been recommended; this allows the surface of the powder to be measured by an optical method, and

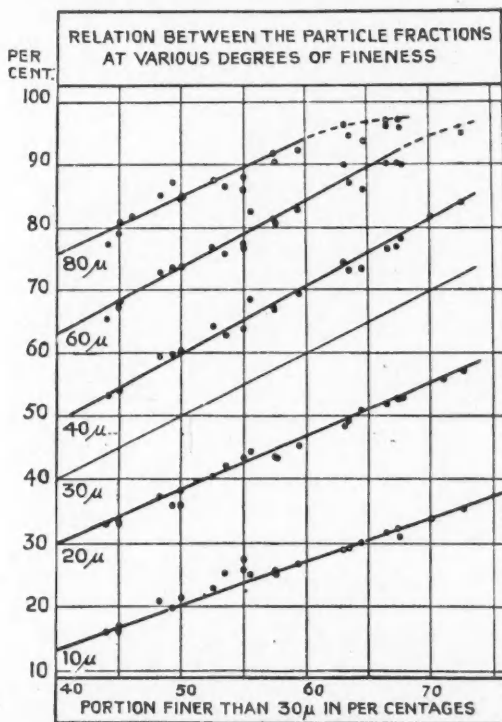


Fig. 2.—Diagram for Obtaining the Particle Size Distribution according to Haegermann.

the Chemische Laboratorium für Tonindustrie has used this method with considerable success. In the meantime, tests have been carried out on a great number of cements so that now a large number of results are available.

The fundamental principle of the optical method is as follows, using the Leukometer (Fig. 3) developed by E. Witte⁶. In this case the whiteness, and hence also the blackness, are measured, and not the tint. The method consists in comparing the brightness by moving a lamp, the distance of which gives the

⁵ *Tonindustrie Zeitung*, 1935, No. 70, p. 845.

⁶ E. Witte, *Glückauf*, 1934, No. 40, p. 923. *Chem. Fabrik*, 1935, Nos. 29 and 30, p. 285.

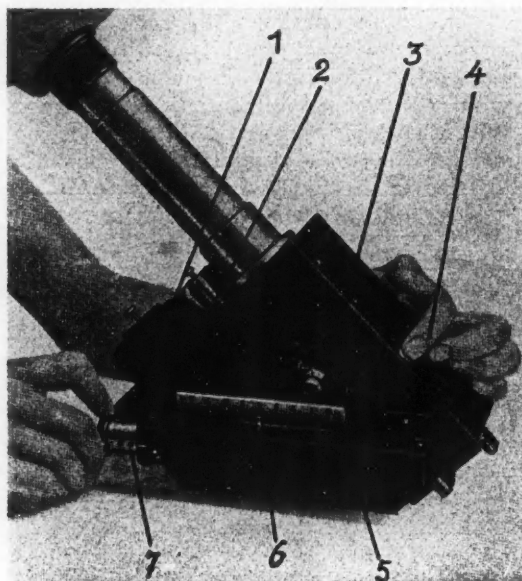


Fig. 3.—Leucometer, according to Witte.

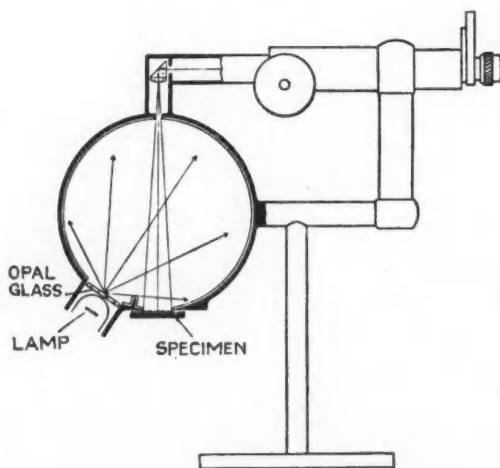


Fig. 4.—Pulfrich Photometer.

critical number. A Baryta Plate (perfect whiteness equals 100) is used as a comparison; the other surface is the material being tested. Both surfaces are illuminated by an electric lamp. The current for the lamp is given by an ordinary flash lamp battery (1). The light is switched on by the push button (4). For prolonged measurements a screw is provided on the two contact studs (2) so that the current can be taken from a bell transformer. The position of the lamp is read by the pointer (6) on the scale (5), and hundredths on the drum (7). For materials under 20 per cent. whiteness content, a neutral grey filter is used; the change over from the bright to the dark range is carried out by the knob (3).

For further comparative tests the sphere reflectometer is used in combination with a Pulfrich photometer by Zeiss (*Fig. 4*). A hollow sphere with a diffusing reflecting white surface is provided on its inside surface. There is an opening at the bottom of the sphere against which the object being tested can be placed. Near this opening a source of light is arranged so that the light shines on the inside of the sphere but cannot shine directly on the test-piece. The test-piece is therefore uniformly illuminated only by the diffused reflected light from the wall of this sphere. The photometer consists essentially of two parallel telescopes with a common eye-piece. The circular field of view is divided into two semi-circles by a vertical cross wire. By turning the measuring drum, the intensity of the field of view can be varied and measured. For the test, the specimen of cement is first examined according to the usual methods with respect to its particle distribution. For instance, the air elutriator of Gonell or the pipette apparatus of Andreasen could be used. The particle fractions obtained are used for calculating the specific surface according to Kühl and Tokune. In order to increase the time of fall during sedimentation the height of fall in the apparatus should be increased. The coarse fractions can be easily determined without using ethylene glycol. Dr. Haegermann placed at the disposal of the author a number of cements which have been examined by his method. In order to obtain comparative values the particle size distribution was determined by sedimentation in an Andreasen pipette which had been lengthened to 35 cm. The surface number was calculated from the whiteness or blackness content by the optical method from the formula

$$K = \frac{W_1 \cdot S_3 - W_3 \cdot S_1}{W_3 \cdot S_2 - W_2 \cdot S_3}$$

This number expresses the relation of the specific surface of the cement to that of the standard powder used, which in this case was powdered coal of approximately cement fineness. The two substances are mixed in equal proportions. W_1 , W_2 and S_1 , S_2 are the whiteness and blackness contents respectively of the components of the mixture (whiteness content + blackness content = $W + S = 100$); W_3 and S_3 are the whiteness and blackness contents of the mixture. K is a coefficient which represents the ratio of the surfaces of the two substances. If the specific surface of this is known then it is immediately possible to calculate the specific surface of the cement.

TABLE 1.—PARTICLE-SIZE DISTRIBUTION OF 6 PORTLAND CEMENTS OBTAINED BY DR. HAEGERMANN'S METHOD AND COMPARED WITH SOME OBTAINED BY ANDREASEN'S METHOD.

| | (1) | | (2) | | (3) | |
|-------|-------------|------------|-------------|------------|-------------|------------|
| | Haegermann. | Andreasen. | Haegermann. | Andreasen. | Haegermann. | Andreasen. |
| μ | Per cent. | Per cent. | Per cent. | Per cent. | Per cent. | Per cent. |
| 0-5 | 16.7 | — | 16.3 | — | 14.2 | — |
| 0-10 | 29.8 | 25.4 | 28.2 | 14.4 | 24.5 | 18.1 |
| 0-20 | 50.0 | 51.4 | 47.6 | 46.7 | 43.7 | 42.1 |
| 0-30 | 63.2 | 65.7 | 61.0 | 58.1 | 56.0 | 52.1 |
| 0-40 | 74.7 | — | 71.7 | — | 66.5 | — |
| 0-60 | 87.5 | — | 84.5 | — | 79.7 | — |
| 0-88 | 98.5 | — | 95.3 | — | 90.1 | — |
| 0-200 | 100.0 | — | 100.0 | — | 100.0 | — |

| | (4) | | (5) | | (6) | |
|-------|-------------|------------|-------------|------------|-------------|------------|
| | Haegermann. | Andreasen. | Haegermann. | Andreasen. | Haegermann. | Andreasen. |
| μ | Per cent. | Per cent. | Per cent. | Per cent. | Per cent. | Per cent. |
| 0-5 | 10.7 | — | 8.5 | — | 7.5 | — |
| 0-10 | 19.4 | 13.4 | 17.0 | 18.4 | 15.7 | 11.1 |
| 0-20 | 34.4 | 34.6 | 33.5 | 34.8 | 32.0 | 33.0 |
| 0-30 | 48.7 | 46.8 | 43.9 | 47.5 | 42.4 | 43.2 |
| 0-40 | 58.0 | — | 52.5 | — | 51.0 | — |
| 0-60 | 72.3 | — | 67.3 | — | 65.6 | — |
| 0-88 | 87.1 | — | 83.0 | — | 78.7 | — |
| 0-200 | 100.0 | — | 100.0 | — | 100.0 | — |

TABLE 2.—COMPARISON OF SPECIFIC SURFACES, IN SQUARE CENTIMETRES PER GRAM, ACCORDING TO HAEGERMANN WITH THE SURFACE FACTORS ACCORDING TO WITTE.

| | 1 | 2 | 3 | 4 | 5 | 6 |
|------------------------|-------|-------|-------|-------|-------|-------|
| Haegermann: | | | | | | |
| 0-5 μ | 2350 | 2135 | 1775 | 1525 | 1360 | 1230 |
| 0-10 μ | 1670 | 1610 | 1463 | 1255 | 1140 | |
| Surface factor: | | | | | | |
| Pulfrich-photometer .. | 0.028 | 0.022 | 0.018 | 0.016 | 0.015 | 0.014 |

TABLE 3.—PARTICLE SIZE ANALYSIS ACCORDING TO GONELL.

| Particle size. | 7 | 8 | 9 | 10 |
|----------------|-----------|-----------|-----------|-----------|
| μ | Per cent. | Per cent. | Per cent. | Per cent. |
| 0-5 | 9.5 | 5.1 | 5.9 | 18.8 |
| 0-10 | 21.8 | 19.2 | 25.7 | 31.3 |
| 0-20 | 31.7 | 35.7 | 41.9 | 49.7 |
| 0-30 | 41.2 | 49.7 | 56.0 | 66.2 |
| 0-40 | 43.8 | 60.6 | 67.5 | 79.0 |
| 0-60 | 89.0 | 80.7 | 78.2 | 94.4 |
| 0-200 | 100.0 | 100.0 | 100.0 | 100.0 |

TABLE 4.—SURFACES CALCULATED FROM TABLE 3 AND SURFACES DETERMINED DIRECTLY ACCORDING TO WITTE.

| | 7 | 8 | 9 | 10 |
|--------------------------------|-------|-------|-------|-------|
| Calculated specific surface .. | 1460 | 1260 | 1470 | 2240 |
| Surface factor : | | | | |
| Witte-Leukometer .. | 0.015 | — | — | 0.024 |
| Pulfrich-photometer .. | 0.013 | 0.012 | 0.017 | 0.026 |

TABLE 5.—COMPARISON OF SPECIFIC SURFACES CALCULATED FROM AIR SEPARATION AND SEDIMENTATION WITH THE SURFACE FACTORS FROM THE OPTICAL METHOD.

| μ | 11 | | | 12 | | |
|------------------------|-------------------------|-------------|------------|-------------------------|-------------|------------|
| | Gonell. | Haegermann. | Andreasen. | Gonell. | Haegermann. | Andreasen. |
| 0—5 | 21.3 | 16.8 | 16.5 | 9.6 | 17.5 | 14.0 |
| 0—10 | 28.7 | 30.4 | 29.2 | 23.9 | 30.4 | 25.4 |
| 0—20 | 48.7 | 50.4 | 48.0 | 42.0 | 50.7 | 46.5 |
| 0—30 | 62.2 | 64.1 | 64.1 | 58.0 | 64.0 | 64.0 |
| 0—40 | 73.3 | 75.8 | 71.8 | 65.2 | 75.7 | 75.4 |
| 0—60 | 80.0 | 88.4 | 83.0 | 91.2 | 88.0 | 86.8 |
| 0—200 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 |
| Gonell | 2600 cm ² /g | | | 1725 cm ² /g | | |
| Andreasen | 2130 .. | | | 1910 .. | | |
| Haegermann : | | | | | | |
| 0—5 μ | 2190 .. | | | 1980 .. | | |
| 0—10 μ | 1710 .. | | | 1590 .. | | |
| Surface Factor : | | | | | | |
| Witte-Leukometer .. | 0.026 | | | 0.016 | | |
| Pulfrich-photometer .. | 0.016 | | | 0.016 | | |

| μ | 13 | | | 14 | | |
|------------------------|-------------------------|-------------|------------|-------------------------|-------------|------------|
| | Gonell. | Haegermann. | Andreasen. | Gonell. | Haegermann. | Andreasen. |
| 0—5 | 6.9 | 16.2 | 10.5 | 24.0 | 19.5 | 20.2 |
| 0—10 | 25.0 | 29.3 | 22.5 | 40.2 | 32.7 | 39.2 |
| 0—20 | 41.9 | 49.2 | 51.7 | 53.0 | 53.5 | 56.8 |
| 0—30 | 61.4 | 62.4 | 62.4 | 62.7 | 67.3 | 67.3 |
| 0—40 | 75.4 | 73.8 | 74.2 | 72.7 | 79.4 | 79.7 |
| 0—60 | 93.9 | 86.3 | 83.8 | 92.7 | 91.5 | 85.0 |
| 0—200 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 |
| Gonell | 1490 cm ² /g | | | 2500 cm ² /g | | |
| Andreasen | 1700 .. | | | 2480 .. | | |
| Haegermann : | | | | | | |
| 0—5 μ | 2130 .. | | | 2400 .. | | |
| 0—10 μ | 1730 .. | | | 1810 .. | | |
| Surface Factor : | | | | | | |
| Witte-Leukometer .. | 0.016 | | | 0.027 | | |
| Pulfrich-photometer .. | 0.016 | | | 0.024 | | |

In Table 1 are given the particle-size distributions, according to Dr. Haegermann, of six Portland cements compared with the results of the sedimentation tests according to Andreasen. Haegermann's values were obtained by the graphical method of estimation from the values of the particle fraction 0 to $30\ \mu$ obtained by sedimentation and the residue on the DIN sieve No. 0.09. The comparison gives a good agreement for the particle fractions 0 to $20\ \mu$ and 0 to $30\ \mu$, but a large divergence for the fraction 0 to $10\ \mu$. The specific surfaces of the cements were calculated from the particle sizes according to Dr. Haegermann, first taking into account the values for 0 to $5\ \mu$ and then neglecting these. This shows the influence which the finest fraction exercises on the surface. In Table 2 these values are set out together with the surface factors obtained by the Pulfrich photometer. In Table 3 the particle-size distribution of three Portland cements (Nos. 7 to 9) and a high-strength cement (No. 10) are set out as obtained by the Gonell air elutriator. The specific surface was calculated and is compared in Table 4 with the figures obtained by the optical method. The cements shown in Table 5 were separated by both air elutriation and by sedimentation and their specific surfaces calculated. From the Andreasen numbers for 0 to $40\ \mu$ the particle fractions (according to Haegermann) were obtained graphically and the specific surface was calculated first for 0 to $5\ \mu$ and then for 0 to $10\ \mu$. Further, the surface number was obtained by the optical method. Specimens Nos. 11 and 12 were special cements, No. 13 an iron Portland cement, and No. 14 a blastfurnace cement. These tests show that there is considerable agreement between the various methods of estimation. They show that, besides the sedimentation and air elutriation methods, the optical method can be used for obtaining the specific surface of cements and that the last is quicker. The deviation in the case of cement No. 11 is being investigated further.

In a note on Dr. Hornke's article H. Elsner von Gronow points out that all the deviations of surface of the finest fraction shown on the left of Table 2 are due to the usual errors of extrapolation to the value 0 as the finest particle size. The extrapolation point should be $3\ \mu$ for the finest fraction in the Haegermann method (see *Ton. Zeit.*, 1932). Tests made according to the Andreasen method have convinced him that particles smaller than $3\ \mu$ in diameter do not occur in Portland cements.

There is a considerable deviation in the specific surfaces of cement published in technical journals in America and Germany. A value of 1,400 square centimetres per gram represents in Germany the surface of a very finely ground cement (obtained with the usual methods of measurement and by standardisation, see *Die Betonstrasse*, 1936, p. 213); but in the U.S.A. this represents a comparatively coarsely ground product (measured by the Wagner turbidimeter).

The method of obtaining the specific surface by integration of the particle size distribution curve is shown in detail in *Chemistry of Cement and Concrete*, by Lea and Desch. This method should be more commonly used and eventually employed for standardising apparatus which has to give the specific surface of cements and fillers by the optical method. When there is international

agreement whether to assume the spherical or cubical shape of mineral particles for calculating the surface of one gram of powder we shall obtain the desired uniformity in the surface results as given in the literature. The first standardisation of the turbidimeter is necessarily based on the assumption of an average particle shape whatever be the optical apparatus used. It should be remembered that the turbidimeter is affected by the colour of the cement grains. This is often overlooked, and naturally does not occur in the case of air elutriation or sedimentation methods.

Recent Patents Relating to Cement.

Refractory Linings for Rotary Furnaces.

454,946. Carborundum Co., Niagara Falls, New York, U.S.A. April 9, 1935.

Rotary cement and lime kilns are provided with linings of cast refractory material having a scratch hardness of about 8 or over. Suitable refractories are cast alumina, either the alpha or beta variety, a material composed of alumina with a minor percentage, e.g. 5 per cent., of magnesium calcium or soda with or without a small percentage of silica, magnesia spinel, a cast chromite alumina fusion, and refractions. These have a melting point higher than 1,800 deg. C. and some have a scratch hardness of about 9.

Cements and Concretes.

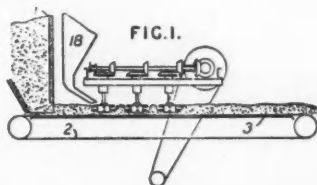
454,944. Dewey & Almy, Ltd., Shell-Mex House, Strand, London. April 9, 1935.

A water-soluble hydroxy-alkyl amine, or a salt or derivative thereof, or a chemical compound or combination which will give rise to the same under concrete mixing conditions, is added to dry hydraulic cement or cement clinker or concrete made from the cement, whereby the compressive strength of the concrete is increased. Triethanol amine, diethanol amine, tri-isopropanol amine, the monocarbonic acid ester of triethanol, the monosodium substitution product of triethanol amine, diethanol carbamide, ethanol isocyanate monoethanol-mono-butanol cyanamide, hydroxy-alkyl ammonium chloride or sulphate and a triethanol ammonium salt of a dispersing agent obtained by condensing sulphonic acids of aromatic hydrocarbons with

formaldehyde may be used. Diatomaceous earth and heavy metal stearates and oleates may be added to the concrete mix.

Cement.

454,961. Middelboe, K., 14, Thorvaldsensvej, Frederiksberg, near Copenhagen. April 11, 1935.



Raw cement material is converted into nodules or a porous mass by being spread in a thin layer on a support and subjected to mixing by an apparatus between which and the support there is relative movement, means being provided for feeding a liquid or dry material as necessary. In the form shown, the raw cement is supplied from a hopper (1) on to an endless band (2) travelling over a plate (3) and is retained by side belts or by fixed walls. The liquid or dry material is supplied from a container (18) and the layer is agitated by a series of high-speed mixing devices consisting of radial arms on a vertical shaft. The material may be turned by a fixed blade arranged between two mixers. In a modification, the mixers travel in a circuitous path over a fixed circular bed, means being provided for feeding and removing the material.

Apparatus for Studying the Effect of Aggressive Solutions on Mortar.

By DR. C. R. PLATZMANN.

THE article by Mr. D. Irvine Watson in the March issue of this journal calls for some comment. The suggested procedure undoubtedly has the advantage that results are obtained in a few days whereas the usual methods take months to perform. The advantages claimed by Mr. Watson for his method certainly do exist. However, this method may give erroneous results for the resistance of mortar or concrete to corrosive solutions. It is generally accepted that the denser and more impermeable the mortar or concrete the better will be its resistance to corrosive solutions. When, according to Mr. Watson's method, the mortar or concrete is pulverised fresh surfaces are exposed which are attacked much more strongly than the unbroken material. This method, therefore, artificially produces the condition of a porous material.

In these circumstances a degree of corrosion and disintegration is obtained which would not exist in the case of unbroken mortar or concrete. If only water, which is not a corrosive solution, is passed through pulverised mortar some of the lime will be dissolved out and a false idea of the resistance will be obtained.

Against these conclusions it may be put forward that when a sufficiently large number of tests is available a factor can be used which will give the behaviour of the material in the unbroken condition. This, however, will apply only for one brand of cement made from the same raw materials in the same way. Since different brands of Portland cement nearly always possess different powers of resistance to corrosive solutions, such a factor is likely to be of doubtful value. For different types of cement such as Portland cement, slag cement, aluminous cement, and pozzolana cement, this factor is useless.

There is a danger that Mr. Watson's method will give results which do not correctly indicate the properties of the cement.

FROM MR. D. IRVINE WATSON.

I am in complete agreement with the comments made by Dr. C. Platzmann, which will, I hope, serve as a warning against any attempt at the present time to extend my suggested use for this apparatus. It is advocated purely for studying the chemical effects produced by aggressive solutions, and not for evaluating the resistance of concrete to attack.

My aim is to induce a number of investigators to attack the purely chemical aspect of the problem on comparable lines in the hope that the knowledge gained may be of assistance in work on the problem of the resistance of concrete as a whole. The latter cannot, of course, be considered out of relation to permeability and several other physical factors, but my apparatus was designed specifically to eliminate these effects to as great a degree as is practicable in a comparatively simple arrangement. So much effort has already been expended in attempts to study the problem under conditions in which all the variables vary, that I consider it time some attempt should be made to come to definite conclusions on the purely chemical aspect of the problem. At present it seems that three months is the minimum length of time for making a test when using this method.

Manufacture of Portland Cement and Sulphuric Acid from Gypsum.

A READER recently asked for references to the literature on the manufacture of Portland cement and sulphuric acid from gypsum, and as this information may be of use to others it is given below.

O. F. Honus. Ger. Pat. No. 47720. Tonind. Ztg., 1929, Vol. 53, p. 949. (Fused cement from gypsum and coal-measure shales.)

Manufacture of Portland Cement and Sulphuric Acid.—G. J. Harris and Imperial Chemical Industries, Ltd. British Patent No. 309661. (Pat. Off. Ill. Official Journal, 1929, Vol. 41 (2107), 2592.)

Manufacture of Portland Cement and Sulphuric Acid.—K. Gordon and Imperial Chemical Industries, Ltd. British Patent No. 309298. (Pat. Off. Ill. Official Journal, 1929, Vol. 41 (2106), 2456.)

Manufacture of Portland Cement and Sulphuric Acid.—C. G. Marks (G. Polysius Akt.-Ges.). British Patent No. 326612. (Pat. Off. Ill. Official Journal, 1930, 42, 2155, 1328.)

Manufacture of Portland Cement from Gypsum. Budnikov and Nekrich.—J. Applied Chem. (U.S.S.R.), 1932, Vol. 5, p. 173. Chem. Abs., 1932, Vol. 26. [(1) The temperature best suited for the decomposition of calcium sulphate is close to its melting point; (2) The CaSO_4 , CaO formed during decomposition also has a low melting point; (3) Carbon oxysulphide is formed in the reducing atmosphere.]

Manufacture of Portland Cement from Gypsum.—Charmandarian and Martschenko. Brit. Chem. Abs. (B), 1934, p. 799. (Mixtures of CaSO_4 , clay, and carbon fuse at less than 1,200 deg. C. owing to corrosion and dissolution of the fine clay; the clinker so obtained yields unsatisfactory cement. Crucibles lined with alumina are refractory and the mass does not fuse after four hours at 1,450 deg. C. The product contains 15 to 25 per cent. SO_3 but no CaS and yields good quality cement.)

Plant for Portland Cement Manufacture from Gypsum.—Budnikoff. Tonind. Ztg., 1934, Vol. 58, 66, 790. (Portland blastfurnace cement plant described. The raw materials are clay, gypsum, and blastfurnace slag.)

Portland Cement Manufacture from Gypsum.—Budnikoff and Jukoff. Tonind. Ztg., 1935, 59, 6, p. 65. (Heat balance calculated for the manufacture of Portland cement from gypsum and clay in a rotary kiln.) Concrete (Cement Mill Edition), 1935, Vol. 43, 3, pp. 40-2; 4, pp. 38-41; and 5, pp. 39-40. (Complete description by Budnikoff.)

SO_2 and Portland Cement from Gypsum.—Rojak, Gerschman, Miloslavski, Nagerova. Brit. Chem. Abs. (B), 1934, p. 16. (Mixture: 2 parts gypsum, 0.37 part clay (at 20 per cent. H_2O), 0.10 part carbon. Fired at 1,450 deg. C. Product is Portland cement clinker with about 1.9 per cent. SO_3 content; the reaction gases are free from H_2S , CO , or COS and have the following average composition: SO_2 , 8.4 per cent.; O_2 , 1.5 per cent.; CO_2 , 21.5 per cent.; N_2 ,

70 per cent.) (An article in *Zeit. Angew. Chemie*, p. 169, 1926, by Muller, describes the plant of Freidr. Bayer and Co. at Leverkusen. Crushed and dried coke and clay slate are mixed and ground with gypsum. This mixture passes through a rotary kiln (164 ft. long and 10 ft. diameter). Pulverised coal is fired in at the lower end of the kiln. The exit gases, after removal of dust, are converted into H_2SO_4 . The monthly output is 3,000 tons of cement clinker and 2,800 tons of SO_2 . In order to obtain a gas of sufficiently high concentration of sulphur dioxide (6 to 7 per cent.) the raw mix must be dried before introducing it into the kiln. The gases must be slightly oxidised since a reducing atmosphere causes the formation of calcium sulphide in the clinker.)

Studies on Mixed Portland Cements.

By KIYOSHI INOUE and SHOICHIRO NAGAI.

A FURTHER report¹ on the authors' studies on high siliceous mixed Portland cements has been published. Many samples of siliceous admixtures were analysed by the methods of ordinary total analysis and special soluble analysis. In the latter method a sample is treated with 10 per cent. NaOH solution and then with 5 per cent. HCl solution to separate the soluble components. The amount of the soluble parts, especially soluble silica, is the most important factor in considering the suitability of the admixture. The results are shown in Table I.

It is seen that the admixtures containing large and medium amounts of soluble silica can be used for mixed Portland cements, but the admixtures containing small amounts of soluble silica do not give good results. For comparison, two samples of blastfurnace slag were analysed after water granulation; these were nearly completely soluble and contained large amounts of lime.

TABLE I.—SOLUBLE ANALYSIS OF VARIOUS SILICEOUS ADMIXTURES.

| Sample No. | Grade of admixture | Loss on ignition | In-soluble part (%) | Soluble parts (%) | | | | | Total soluble part (%) |
|------------|--------------------|------------------|---------------------|-------------------|-------------------------|-------------------------|-------|-------|------------------------|
| | | | | SiO_2 | Al_2O_3 | Fe_2O_3 | CaO | MgO | |
| 277 | High grade .. | 5.54 | 12.20 | 78.45 | 2.16 | 0.20 | 0.43 | 0.07 | 81.31 |
| 278 | Medium grade .. | 7.75 | 27.60 | 54.91 | 8.52 | 0.60 | 0.55 | Trace | 64.58 |
| 407 | " " " | 5.56 | 31.68 | 49.38 | 9.27 | 0.93 | 2.09 | 0.52 | 62.19 |
| 422 | " " " | 13.53 | 20.87 | 50.75 | 8.67 | 1.03 | 2.55 | Trace | 63.00 |
| 477 | " " " | 14.11 | 21.86 | 49.49 | 9.52 | 0.54 | 2.48 | 0.40 | 62.43 |
| 432 | " " " | 14.49 | 25.19 | 46.29 | 8.89 | 0.35 | 2.38 | 0.65 | 56.20 |
| 318 | Low " " " | 3.02 | 52.73 | 36.36 | 6.45 | 0.58 | 0.45 | 0.11 | 43.95 |
| 343 | " " " | 3.82 | 53.70 | 32.24 | 7.06 | 0.98 | 0.93 | 0.36 | 41.59 |
| 442 | " " " | 3.58 | 51.38 | 35.22 | 6.10 | 1.16 | 0.09 | Trace | 43.38 |
| 346 | " " " | 1.15 | 55.02 | 34.98 | 6.62 | 1.24 | 0.78 | 0.52 | 44.15 |
| 320 | Lowest " " " | 3.21 | 72.83 | 17.65 | 5.04 | 0.08 | 0.54 | 0.57 | 23.88 |
| 322 | " " " | 2.66 | 67.68 | 24.42 | 4.22 | 1.04 | 1.64 | 0.21 | 31.53 |
| 340 | " " " | 1.90 | 67.60 | 23.38 | 4.94 | 0.66 | 0.82 | 0.76 | 30.56 |
| 302 | Blastfurnace slag | 0.63 | 0.16 | 31.65 | 14.53 | 0.37 | 43.72 | 5.07 | By total analysis |
| 303 | " " " | 2.86 | 0.19 | 25.10 | 12.93 | 0.51 | 48.25 | 8.67 | |

Many samples of mixed Portland cements were prepared from these admixtures and cement (or clinker) in the proportions of 30 to 45 parts of admixture and

¹ Abstracts of previous reports are given in this journal for February, 1936.

TABLE 2.

| Ser-ies | No. | Kind | Proportions (%) | | | Specific gravity | Fineness Residue on 4900m/ cm-sieve (%) | Loss on igni-tion (%) | In-soluble residue (%) | SiO ₂ (%) | Al ₂ O ₃ (%) | Fe ₂ O ₃ (%) | CaO (%) | MgO (%) | SO ₃ (%) | Sulphide sulphur (%) | Free lime (%) |
|---------|-----|--------------------|-------------------|--|--------|------------------|---|-----------------------|------------------------|----------------------|------------------------------------|------------------------------------|---------|---------|---------------------|----------------------|---------------|
| | | | Cement or clinker | Admixture | Gypsum | | | | | | | | | | | | |
| I | 280 | Portland .. | Clinker (a) 100 | — | 3.0 | 3.10 | 3.0 | 2.16 | 3.19 | 20.78 | 5.76 | 3.34 | 63.44 | 1.99 | 1.61 | — | 1.54 |
| | 281 | Mixed .. | " 60 | No. 278 40 | 2.5 | 2.79 | 1.4 | 4.51 | 31.72 | 41.40 | 7.62 | 2.69 | 40.53 | 1.41 | 1.52 | — | 1.26 |
| | 282 | Mixed .. | " 60 | No. 277 40 | 2.5 | 2.60 | 1.0 | 4.84 | 32.34 | 44.45 | 5.55 | 2.32 | 39.08 | 1.45 | 1.42 | — | 1.15 |
| II | 297 | Portland .. | Cement (A) | — | 2.9 | 3.12 | 2.9 | 0.82 | 0.47 | 21.76 | 5.80 | 2.52 | 65.35 | 1.49 | 1.72 | — | 1.85 |
| | 298 | Mixed .. | " 55 | No. 277 45 | 1.0 | 2.76 | 0.6 | 5.23 | 35.57 | 48.80 | 4.76 | 1.84 | 37.05 | 1.00 | 1.46 | — | 0.55 |
| | 299 | Mixed .. | " 55 | (No. 277 35) (Ca(OH) ₂ 10) | 1.0 | 2.75 | 0.3 | 7.35 | 28.91 | 41.75 | 4.24 | 1.74 | 43.33 | 1.14 | 1.20 | — | 6.77 |
| IV | 334 | Portland .. | Cement (C) | — | 1.0 | 3.11 | 1.0 | 1.65 | 0.34 | 21.34 | 5.29 | 2.49 | 66.28 | 0.92 | 1.46 | — | 2.16 |
| | 335 | Mixed .. | " 75 | No. 320 25 | 1.0 | 2.89 | 0.9 | 3.70 | 20.25 | 31.50 | 6.77 | 2.49 | 51.46 | 0.84 | 1.44 | — | 1.07 |
| | 336 | Mixed .. | " 60 | No. 320 40 | 1.0 | 2.78 | 0.5 | 1.85 | 30.73 | 37.71 | 7.12 | 2.51 | 44.31 | 0.89 | 1.47 | — | 0.70 |
| V | 300 | Blastfurnace slag. | Cement (A) 55 | (No. 302 35) (Ca(OH) ₂ 10) | 1.0 | 2.83 | 2.5 | 4.42 | 0.45 | 22.18 | 6.93 | 1.66 | 58.80 | 2.57 | 1.42 | 0.74 | 8.59 |
| | 301 | " " | " 55 | No. 302 45 | 1.0 | 2.86 | 5.3 | 3.51 | 0.25 | 25.48 | 8.23 | 1.55 | 55.27 | 2.87 | 1.52 | 0.40 | 1.54 |
| | 310 | " " | " 55 | (No. 303 35) (Ca(OH) ₂ 10) | 1.0 | 2.90 | 2.1 | 4.17 | 0.38 | 21.40 | 6.91 | 1.83 | 59.40 | 3.74 | 1.95 | 0.30 | 6.27 |
| VI | 311 | " " | " 55 | (No. 303 45) (Ca(OH) ₂ 10) | 1.0 | 2.95 | 5.7 | 1.75 | 0.40 | 22.44 | 9.58 | 1.67 | 57.60 | 4.66 | 1.53 | 0.72 | 1.70 |
| | 425 | Portland .. | Cement (F) | — | 3.3 | 3.15 | 3.3 | 0.93 | 0.42 | 21.91 | 4.45 | 3.41 | 63.20 | 1.49 | 1.49 | — | 1.06 |
| | 426 | Mixed .. | " 60 | No. 424 40 | 1.0 | 2.69 | 3.1 | 6.02 | 27.19 | 39.00 | 7.07 | 1.85 | 41.24 | 0.96 | 1.47 | — | 0.61 |
| X | 427 | Mixed .. | " 60* | (No. 424 40) (CaCO ₃) | 1.0 | 2.79 | 3.0 | 3.51 | 19.46 | 35.70 | 7.71 | 2.16 | 45.75 | 1.24 | 1.65 | — | 4.08 |
| | 428 | Mixed .. | " 70 | No. 424 30 | 1.0 | 2.85 | 4.4 | 3.91 | 26.24 | 34.79 | 7.87 | 2.13 | 46.80 | 1.07 | 1.31 | — | 0.83 |
| | 429 | Mixed .. | " 70* | (No. 424 30) (CaCO ₃) | 1.0 | 2.89 | 4.4 | 4.45 | 18.86 | 31.44 | 6.53 | 2.42 | 49.70 | 1.22 | 1.52 | — | 2.04 |
| XIV | 307 | Aluminous .. | — | — | 13.8 | 3.27 | 13.8 | 0.27 | 0.73 | 2.31 | 40.42 | 17.25 | 37.40 | 0.80 | Trace | — | — |
| | 350 | " " | " | — | 11.0 | 3.23 | 11.0 | 0.07 | 0.97 | 5.58 | 38.69 | 16.80 | 38.00 | 0.82 | 0 | Trace | — |

* Calculated at 900 deg. C. for two hours.

70 to 55 parts of cement or clinker by grinding with a small amount of gypsum in a small ball mill. The physical properties and chemical compositions of these samples were compared, and some of the results are given in Table 2. From these results it is seen that (1) all mixed Portland cements have lower specific gravities and slower setting times than common Portland cements; (2) chemical compositions of mixed Portland cements differ from common Portland cements, containing large amounts of silica and insoluble residue and small amounts of lime; (3) blastfurnace slag cements differ from other kinds of siliceous mixed Portland cements in the smaller amounts of silica and insoluble residue, larger amounts of alumina and lime, and the proportion of sulphide, sulphur, manganese oxide, etc.; (4) two samples of imported aluminous cement are quite different from the common and mixed Portland cements in their large content of alumina. Some samples of mixed Portland cements, i.e. Nos. 299, 300, 310, 427 and 429, are special mixed Portland cements obtained by mixing and grinding clinker or cement with admixture and slaked lime, or calcined (at about 900 deg. C. for two hours) mixtures of admixture and limestone; these special mixed Portland cements have setting times similar to those of common Portland cement owing to the free lime added.

The samples were tested for mortar strengths with the dry or non-plastic mortar of the Japanese Standards for Portland Cement and Blastfurnace Slag Cement and with the wet or plastic mortar proposed by Professor Rös in Switzerland and by Dr. Haegermann in Germany. High siliceous mixed Portland cements, i.e. Nos. 282, 298, 299, etc., give remarkably high strengths with non-plastic mortar (greater than those of aluminous cements, high-early-strength Portland cements, etc.) but their strengths with plastic mortars are smaller than those of common Portland cement.

The samples were tested for expansion, contraction, and corrosion, with prismatic test pieces (4 by 4 by 16 cm.) of plastic mortar cured in water, in 10 per cent. Na_2SO_4 solution and in 10 per cent. NaCl solutions for 24 weeks. Common Portland cements expanded and were strongly attacked by the 10 per cent. Na_2SO_4 solution and cracked and disintegrated after eight weeks in 10 per cent. Na_2SO_4 solution. They were followed by aluminous cements, blastfurnace slag cements, and mixed Portland cements from low-grade admixtures in this order.

The research is being carried out at the Institute of Silicate Industry, Department of Applied Chemistry, Faculty of Engineering, Tokio Imperial University.

Trade Notice.

PNEUMATIC CONVEYORS.—An illustrated catalogue of 64 pages describing "Sirocco" pneumatic conveying plant has been issued by the manufacturers, Messrs. Davidson & Co., Ltd., of Belfast. The system should be of special interest to cement and hydrated lime makers in the handling of their finished products and powdered coal over distances up to 1,000 ft. horizontally and 150 ft. vertically. Copies of the catalogue will be sent on request to those interested.